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Structure of 4,4′,5,5′-Tetranitro-2,2′-biimidazole Dihydrate*

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Abstract. $C_6H_2N_8O_8.2H_2O$, $M_r = 350{\cdot}16$, monoclinic, $P2_1/n$, $a = 5{\cdot}031\ (2)$, $b = 8{\cdot}478\ (1)$, $c = 15{\cdot}362\ (1)$ Å, $\beta = 97{\cdot}18\ (2)°$, $V = 650{\cdot}1$ Å^3, $Z = 2$, $D_x = 1{\cdot}789$ Mg m^{-3}, λ(Mo $K\alpha_1$) $= 0{\cdot}70926$ Å, $\mu = 0{\cdot}16$ mm^{-1}, $F(000) = 316$, room temperature, final $R = 0{\cdot}034$ for 859 observed reflections [$I > 2\sigma(I)$] out of 1133 independent reflections. The molecule lies on a center of symmetry. The hydrogen on the ring nitrogen is strongly hydrogen bonded to the water oxygen [N···O = 2·656 (3) Å], but the water hydrogens are only weakly bonded to the nitro-group O atoms [O···O = 3·137 (4), 3·073 (3) Å]. There are no unusual intramolecular distances.

Experimental. Title compound prepared by nitration of 2,2′-biimidazole by $NaNO_3$ in concentrated H_2SO_4. Crystals grown by evaporation of water solution. Selected crystal *ca* 0·43 × 0·27 × 0·16 mm. CAD-4 diffractometer, θ–2θ scan. Scan range $(1{\cdot}0 + 0{\cdot}34\tan\theta)°$. Scan speed 1·0 to 5·5° min^{-1}. Background first and last 1/6 of scan. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell, 25 reflections $12 < \theta < 24°$. No absorption corrections. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}596$ Å^{-1}. Index range $-5 \le h \le 5$, $0 \le k \le 10$, $-18 \le l \le 18$, 1635 reflections measured and averaged to yield 1138 unique reflections of which 859 were observed with $I > 2\sigma(I)$, merging $R_F = 0{\cdot}010$. Standard reflections $\bar{1}55$ and $1\bar{4}6$ showed no significant variation. Least-squares refinement minimized $\sum w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0{\cdot}03F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971), hydrogens by difference Fourier. Scale factor, isotropic type II extinction parameter (Larson, 1969), positional parameters, anisotropic thermal parameters for C, N, O, and isotropic thermal parameters for H were refined. Final $R = 0{\cdot}034$, $wR = 0{\cdot}046$, $S = 2{\cdot}1$. Max. $\Delta/\sigma = 0{\cdot}05$. Final ΔF Fourier synthesis $-0{\cdot}16 < \Delta\rho < 0{\cdot}19$ e Å^{-3}. Scattering factors f (RHF for C, N, O and SDS for H), f', f'' from *International Tables for X-ray Crystallography* (1974). Calculations on CRAY-1 using the Los Alamos crystal structure system developed primarily by A. C. Larson.

Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the molecule showing the atomic numbering scheme.

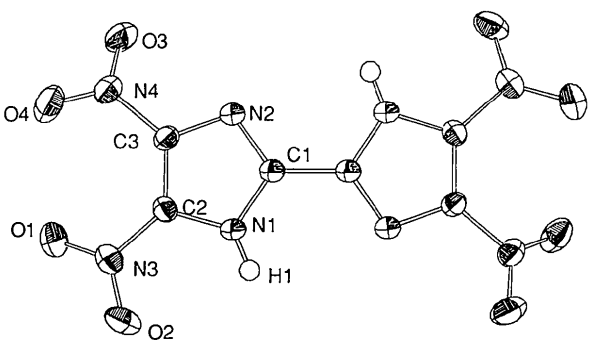

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule to show the atomic numbering scheme. Thermal ellipsoids are 30% probability. Hydrogen size is arbitrary.

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0108-2701/90/101957-02$03.00

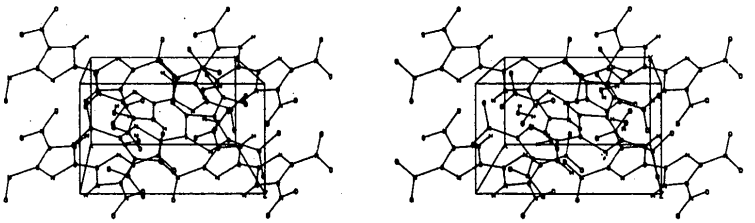

Fig. 2. Stereo drawing of the structure. The origin is at the upper left rear.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^2$) *with e.s.d.'s in parentheses*

$$U = (1/3)\sum_i U_{ii}.$$

	x	y	z	U_{eq}
C(1)	5340 (4)	4343 (2)	0290 (1)	3·8 (2)
C(2)	7490 (4)	2926 (3)	1311 (1)	4·0 (2)
C(3)	5299 (4)	2126 (2)	0903 (1)	4·1 (2)
N(1)	7482 (3)	4344 (2)	0912 (1)	3·9 (2)
N(2)	3958 (3)	3003 (2)	0274 (1)	4·1 (2)
N(3)	9451 (4)	2564 (2)	2036 (1)	5·1 (2)
N(4)	4419 (4)	0528 (2)	1041 (1)	5·1 (2)
O(1)	9027 (4)	1471 (2)	2513 (1)	7·5 (2)
O(2)	11442 (4)	3411 (2)	2140 (1)	6·7 (2)
O(3)	2033 (4)	0276 (2)	0879 (1)	8·1 (2)
O(4)	6086 (4)	−0460 (2)	1289 (1)	7·8 (2)
Water				
O(5)	4093 (4)	1741 (2)	3918 (1)	6·3 (2)

Type II isotropic extinction parameter = 1·7 (5) × 10^{-5} mm (Larson, 1969).

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(1)	1·441 (1)	C(3)—N(2)	1·334 (3)
C(1)—N(1)	1·347 (3)	C(3)—N(4)	1·449 (3)
C(1)—N(2)	1·331 (3)	N(3)—O(1)	1·216 (2)
C(2)—C(3)	1·377 (3)	N(3)—O(2)	1·227 (3)
C(2)—N(1)	1·349 (3)	N(4)—O(3)	1·214 (3)
C(2)—N(3)	1·427 (3)	N(4)—O(4)	1·213 (3)

C(1)—C(1)—N(1)	123·1 (1)	N(2)—C(3)—N(4)	119·4 (2)
C(1)—C(1)—N(2)	124·4 (1)	C(1)—N(1)—C(2)	106·1 (2)
N(1)—C(1)—N(2)	112·6 (2)	C(1)—N(2)—C(3)	104·3 (2)
C(3)—C(2)—N(1)	106·0 (2)	C(2)—N(3)—O(1)	118·6 (2)
C(3)—C(2)—N(3)	133·3 (2)	C(2)—N(3)—O(2)	116·7 (2)
N(1)—C(2)—N(3)	120·6 (2)	O(1)—N(3)—O(2)	124·7 (2)
C(2)—C(3)—N(2)	111·0 (2)	C(3)—N(4)—O(3)	116·6 (2)
C(2)—C(3)—N(4)	129·5 (2)	C(3)—N(4)—O(4)	118·7 (2)
		O(3)—N(4)—O(4)	124·8 (2)

Hydrogen-bond geometry

X—H···Y	X—Y (Å)	H···Y (Å)	X—H···Y (°)
N(1)—H(1)···O(5)^{i}	2·656 (3)	1·83 (2)	171 (2)
O(5)—H(2)···O(3)^{ii}	3·073 (3)	2·32 (3)	140 (3)
O(5)—H(3)···O(2)^{iii}	3·215 (3)	2·49 (3)	158 (4)

Symmetry operations: (i) $3/2 - x$, $1/2 + y$, $1/2 - z$; (ii) $x - 1$, y, z; (iii) x, y, z.

Final parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Stereo drawing shown in Fig. 2. The asymmetric unit is 1/2 molecule of $C_6H_2N_8O_8$ and one molecule of water. The rings are planar to within 0·03 Å. The nitro groups are twisted out of the ring plane by 17·1 and 31·6°, the group with the smaller twist being adjacent to the H atom. In biimidazole itself (Cromer, Ryan & Storm, 1987) the molecules are joined into ribbons by pairs of hydrogen bonds on each side of the molecule. In the present case the nitro groups probably prevent the molecules from approaching close enough to form these hydrogen bonds. The next best thing is to bond to a solvent molecule. Bond lengths are close to those found in biimidazole and the diammonium salt of tetranitrobiimidazole (Cromer & Storm, 1990).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving hydrogen have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52527 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. References to structures of other small, high-energy molecules are given by Cromer, Hall, Lee & Ryan (1988).

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